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**Cherman : D.J. Ward (Australia)**

**Secretary General: W.H. Lakin (IWTO)**

**Techn. Coordinator: G. Mercier (France)**

**Report Nrº 3**

**TITLE: "Influence of liposome on wool dyeing kinetics"**

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### SUMMARY OF FINDINGS:

This study describes the role of commercial liposomes as dyeing auxiliaries in wool dyeing with Lanaset Dyes. Dye exhaustion, colour fastness, fabric handle measurements and compatibility of dyes are studied in different dyeing kinetics with Albegal SET, liposome (Ecotrans W-8814), and without any auxiliary. The use of commercially available liposomes favours dye exhaustion improving dye levelling, colour fastness and handle characteristics of dyed fabrics. The use of liposomes in wool dyeing with Lanaset Dyes promotes energy saving and ecological benefit.

### COMMERCIAL IMPLICATIONS – CURRENT & FUTURE:

Over the last decade, a number of investigations have been carried out using different carriers capable of reducing the degradative effect brought about in conventional wool dyeing. Thus, the technology of microencapsulation has given rise to a number of innovations utilising the basic principles of targeting, slow release and protection of this sensitive fibre. The potential use of liposomes as carriers in wool finishing is based on (i) the similarity existing between the bilayer structure of the "cell membrane complex" (CMC) and that of liposomes, (ii) the important role played by the CMC in the transport of chemicals into the fibres and (iii) the relevant importance of the hydrophobic interactions in the structural organisation of wool.

At present there is a commercial liposome in the textile market, Ecotrans W-8814 with competitive cost to conventional auxiliaries. This new dyeing auxiliary controls the rise of the colour giving good levelness with the advantage to promote higher dye exhaustion than other synthetic auxiliaries. Furthermore, the final temperature of the dye process can be reduced on 10°C that will greatly favour the handle characteristics of the fibre with an energy saving. The biological nature of the liposome is an important ecological benefit of this new dyeing process.

Industries and Research Centers from Italy, Portugal and Spain support this innovation participating in a European project to demonstrate the industrial feasibility of this dyeing process.

Chairman: D.J. Ward (Australia)

Secretary General: W.H.Lakin (IWTO)

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## INFLUENCE OF LIPOSOMES ON WOOL DYEING KINETICS

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## SUMMARY

This study describes the role of commercial liposomes as dyeing auxiliaries in wool dyeing with Lanaset dyes. Dye exhaustion, colour fastness, fabric handle measurements and compatibility of dyes are studied in different dyeing kinetics with Albegal SET, liposome (Ecotrans W-8814), and without any auxiliary. The use of commercially available liposomes favours dye exhaustion improving dye levelling, colour fastness and handle characteristics of dyed fabrics. The use of liposomes in wool dyeing with Lanaset dyes promotes energy saving and ecological benefit.

## INTRODUCTION

Liposomes are vesicle structures formed by surface-active biological lipids. They can be prepared as small unilamellar vesicles (SUV), multilamellar vesicles (MLV) or large unilamellar vesicles (LUV). In all cases, an internal aqueous domain is entrapped between lipidic bilayers. Phosphatidylcholine is the most widely biological lipid used in the preparation of liposomes.

The vehicle capacity of the liposomes has been explored in different fields (pharmaceutical, cosmetic, etc.) with interesting and technological useful results. The technology of microencapsulation has given rise to a number of innovations in the textile domain, using the basic principles of targeting, slow release and protection of fibrous material [1]. The potential use of liposomes as carriers in wool finishing has been widely demonstrated in the laboratory [2, 3].

Traditionally, in the textile field high temperature procedures (about 100°C or more) and dyeing auxiliaries (synthetic compounds) have been widespread used. In both cases, the deleterious effects for textiles (fibre damage) and for the environment (contamination) are quite obvious. The clear reduction of the temperature of the wool dyeing process using liposomes obtained by our group (about 10°C) with the corresponding energy saving, the ecological benefits of the process (avoiding the use of synthetic auxiliary products) as well as the final quality of the textile dyed are the main advantages of this process.

A preliminary industrial feasibility of the process was demonstrated by an Exploratory Phase of a Pre-CRAFT Project. From the results obtained on dye exhaustion, dye fixation and textile handle, it can be deduced an acceptable role for the liposomes in order to avoid the use of synthetic textile auxiliaries in the dyeing process. To specifically demonstrate the competitive cost of liposomes, a complementary research was carried out with fruitfully results obtaining liposomes at a similar cost to synthetic dyeing auxiliaries. At present, we try to obtain more precise information about the intimate mechanism involved in the previous dye-liposome interactions and in the subsequent steps of the wool dyeing process.

The present work deals with a practical approach of this new wool dyeing process using commercial liposomes and Lanaset dyes. Since these dyes give good compatibility when applied with the auxiliary Albegal SET, a kinetic study was planned comparing the use of liposomes and Albegal SET as alternative auxiliaries in the wool dyeing process.

## **EXPERIMENTAL**

### **Materials**

Wool 100% yarn 31.25tex (1/32Nm) and botany wool fabrics knitted from R64/2 tex (count 2/28) yarns were used. The commercially available Ciba-Geigy Lanaset Red G, Lanaset Grey G, Lanaset Brown B, Lanaset Yellow 2R and Albegal SET were selected given their widespread use in wool dyeing at an industrial scale. The commercially available liposome for textile applications Ecotrans W-8814 containing 20% of lipids was supplied by CRESA (Barberà del Vallès, Spain). The non-ionic surfactant Triton X-100 (octylphenol with 10 units of ethylene oxide and active matter of 100%) was supplied by Tenneco S.A. (Barcelona, Spain).

### **Methods**

A deing kinetic study was performed to know the liposomes suitability in the industrial process.

**Table I. Kinetic dyebath compositions.**

	<b>DYES</b>				<b>AUXILIARIES</b>	
	YELLOW LANASET 2R (% owf)	RED LANASET G (% owf)	GREY LANASET G (% owf)	BROWN LANASET B (% owf)	ECOTRANS W-8814 (% owf)	ALBEGAL SET (% owf)
<b>1a</b>	0.5	0.5	0.5	-	0.8	-
<b>1b</b>	0.5	0.5	0.5	-	-	1
<b>2a</b>	1	-	-	-	4	-
<b>2b</b>	1	-	-	-	-	1
<b>2c</b>	1	-	-	-	-	-
<b>3a</b>	-	1	-	-	4	-
<b>3b</b>	-	1	-	-	-	1
<b>3c</b>	-	1	-	-	-	-
<b>4a</b>	-	-	1	-	4	-
<b>4b</b>	-	-	1	-	-	1
<b>4c</b>	-	-	1	-	-	-
<b>5a</b>	-	-	-	1	4	-
<b>5b</b>	-	-	-	1	-	1
<b>5c</b>	-	-	-	1	-	-

Dyeing was initiated at room temperature and the temperature was raised by 1°C/min to 90°C (kinetic with liposome) or 98°C (kinetic with Albegal SET). Dyeing was additionally continued for 30 minutes.

Another series of kinetics was performed with wool knitted fabric samples with every single Lanaset dye in the presence of liposomes (2a, 3a, 4a and 5a) or Albegal SET (2b, 3b, 4b and 5b) or in the absence of auxiliaries (2c, 3c, 4c and 5c). Dyeing was initiated at room temperature and the temperature was raised by 1°C/min to 90°C. Dyeing was additionally continued for 30 minutes.

The compatibility of three Lanaset dyes was studied by visual assessing and using a Macbeth Spectrophotometer. The CIELab coordinates (a, b, L) were obtained.

Colour fastness were analysed with fabric knitted samples. The IWS Test Method n° 193 was applied for determining colour fastness to washing of wool dyed fabrics.

Fabric handle measurements are mainly based on multidirectional bending rigidity, compression behaviour and surface friction tests. The knitted fabric structure has a prominent effect in bending rigidity and compression, the influence of the auxiliary used could not be pointed out. Only the surface friction showed a slight relationship with the type of the dyeing auxiliary used.

The surface friction is likely to have a significant impact upon the assessment of fabric handle. Depending on the friction surface level a comfortable and consistent feeling could be obtained

according to the level of modification of static and dynamic surface friction induced by the auxiliary.

The standard method of fabric and dynamic surface friction is to pull a sledge of known dimensions and weight over the sample at constant speed. The force to overcome friction is measured and the coefficient of friction determined. The force that causes the initial sliding between surfaces is the static friction, while the force to maintain the sliding state up to the end of the test is the dynamic friction.

Sledge was covered by a sample of the same fabric; therefore measurements of fabric to fabric surface friction were obtained. Sledge size was 60×60 mm, the weight over the sample was 200 cN and the sliding speed was 100 mm/min along 80 mm. Fabric surface friction was measured on both fabric sides, that is fabric face to face and fabric back to back sides.

## RESULTS AND DISCUSSION

Previous works were focused on the optimisation of the dyeing process varying the amount of liposome and the final temperature using also Lanaset dyes [6,7]. Therefore, in this study we decided to perform a kinetic study with a three different Lanaset dyes and with 0.8% of liposome until a final temperature of 90°C. This kinetic was compared with the industrial process commonly used with these dyes, in the presence of a 1% of Albegal SET until a final temperature of 98°C.

The different dyed samples and residual dyeing baths allowed us to analyse the dyeing processes studying the effects of the different auxiliaries on the kinetic behaviour and on the dye compatibility. A good controlled dye absorption for the two auxiliaries can be seen in Figure 2, although a better control of the dye rise can be observed in the case of liposome at lower temperatures; however, at temperatures higher than 87°C, the liposomes achieve higher dye exhaustion even though the final temperature is lower (90°C) than Albegal SET (98°C).

The dye compatibility was performed by a visual and spectrophotometrical study of the wool samples subjected at the different times and temperatures of the same dyeing processes.

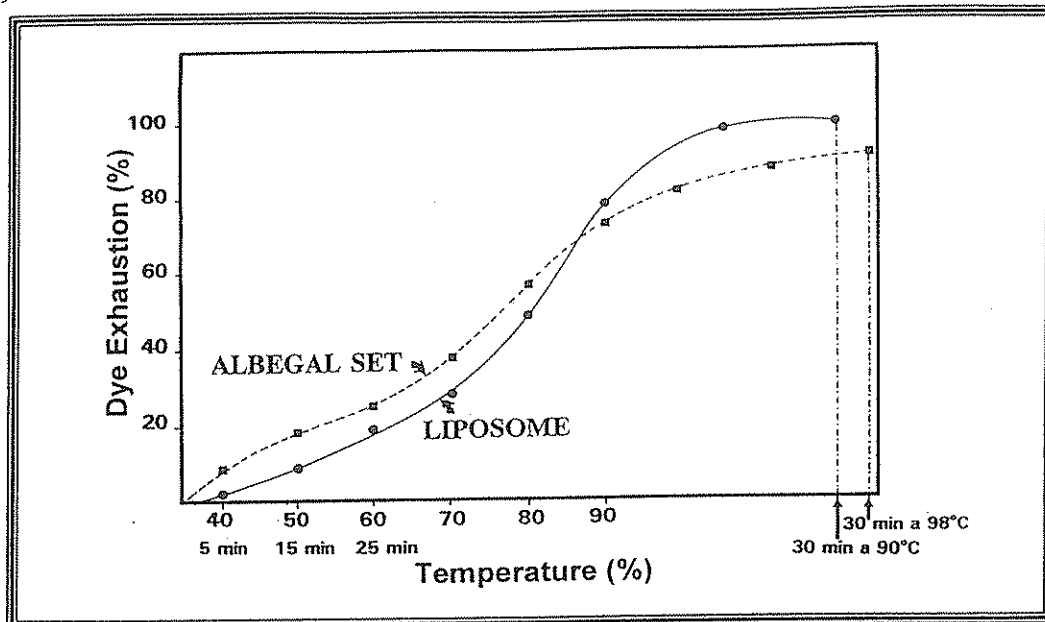


Figure 2: Wool Yarn Kinetics with three different Lanaset dyes.

The spectrophotometric study can be summarised in Figure 3. Eventhough the different behaviour of the two dyeing processes, the compatibility is maintained. In the liposome dyeing process there is a variation of 3.3 units in the  $a$  coordinate, while in the case of Albegal SET dyeing process there is a variation of 3.2 units in the  $b$  coordinate. A final value of 24.43 units in coordinate  $L$  was obtained in the process with liposomes and 26.49 units in the case of the Albegal SET process. These results confirm the higher dye exhaustion values obtained in the colorimetric assessment of the residual dyeing bath in the case of the liposome process and using a similar surface of dyeing level.

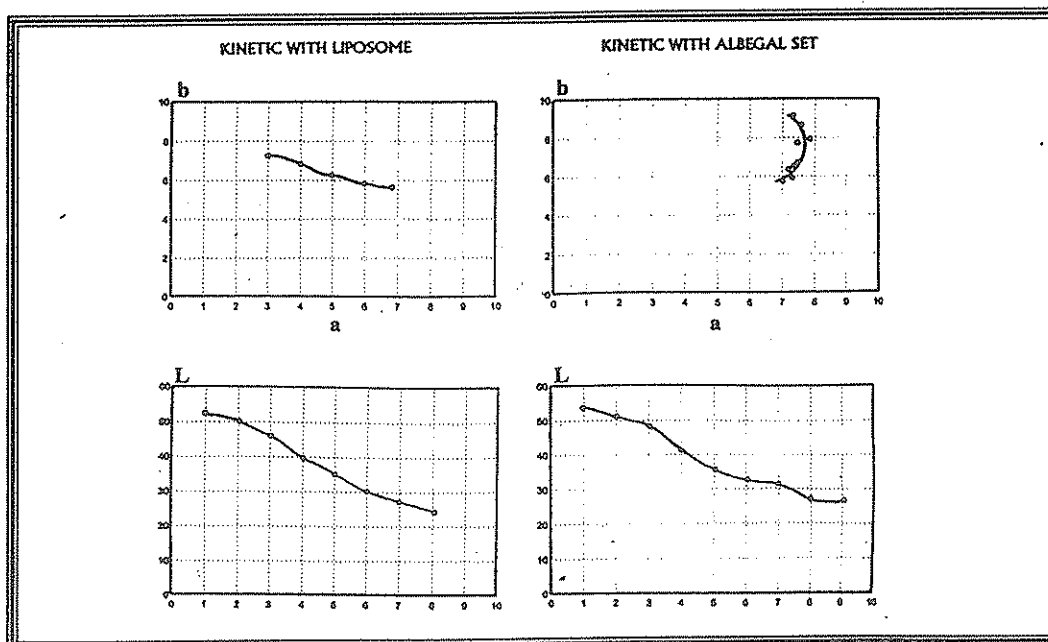


Figure 3: L, a, b representation of wool yarn samples of kinetics (in Fig. 1).

The different rise of the colours obtained in each dyeing process gave us to study separately the kinetic behaviour of each dye in the absence of auxiliary, in the presence of Albegal SET and in the presence of liposome at higher concentration (4% owf) to determine the effect of the liposomes at different temperatures.

In Figure 4, the kinetic behaviour of the four Lanaset dyes applied under the three processes described in the experimental part can be observed. In all cases there is a retention of the colour due to the presence of liposomes or Albegal SET during the first stages of the dyeing process, being more marked in the case of Brown Lanaset B and Grey Lanaset G. Eventhough Yellow Lanaset 2R is better retained with Albegal SET, it is important to emphasise the highest final dye exhaustion obtained for Red Lanaset G and Brown Lanaset B when liposomes are present with a good retention of the dye in the first stages of the process.

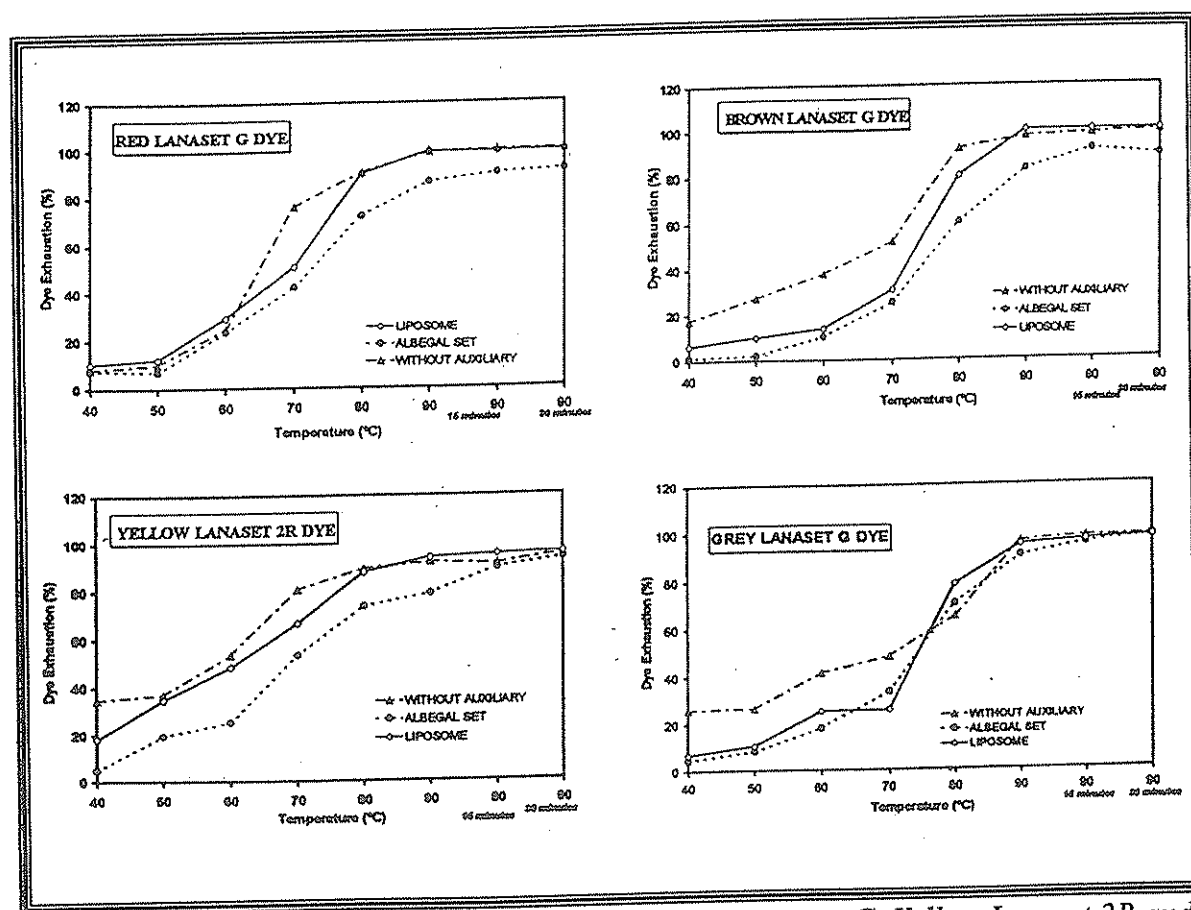


Figure 4: Kinetic representations of Red Lanaset G, Brown Lanaset G, Yellow Lanaset 2R and Grey Lanaset G Dyes.



Dyeing kinetics with individual dyes are in concordance with results previously obtained with these dyes. The higher variation in the a coordinate (green-red axis) in liposome kinetic (Figure 3) is in concordance with the higher retarding effect of liposomes on the Grey Lanaset G dye kinetics (Figure 4).

Furthermore, the higher variation in the b coordinate (blue-yellow axis) in the Albegal SET kinetic (Figure 3) are reflected in kinetic of Figure 4 in which the Yellow Lanaset 2R dye is more retarded with Albegal SET than with liposomes. The different kinetic behaviour of each dye explains the different tones in the rise of colour due to the presence of each auxiliary on the dye bath.

Colour fastness was studied with the dye fabrics of the last stage of the different kinetics using or not the auxiliaries in the different processes with Red Lanaset G, Brown Lanaset B and Yellow Lanaset 2R. The results obtained using the methodology detailed in the experimental part are listed in Table II.

*Table II. Results of IWS Test Method n° 193 on dyed wool samples.*

SAMPLE	ASSESSING STAINING					
	wool	acrylic	polyester	polyamide	cotton	diacetate
2a	5	5	5	4	4-5	5
2b	4-5	5	5	4	5	5
2c	4-5	5	5	4	5	5
3a	3-4	5	4-5	3	4-5	4-5
3b	3	4-5	4-5	3	4-5	4-5
3c	2-3	5	4	2-3	4	4-5
5a	4-5	5	5	4	5	5
5b	4-5	5	5	4	5	5
5c	4-5	5	5	3-4	5	5

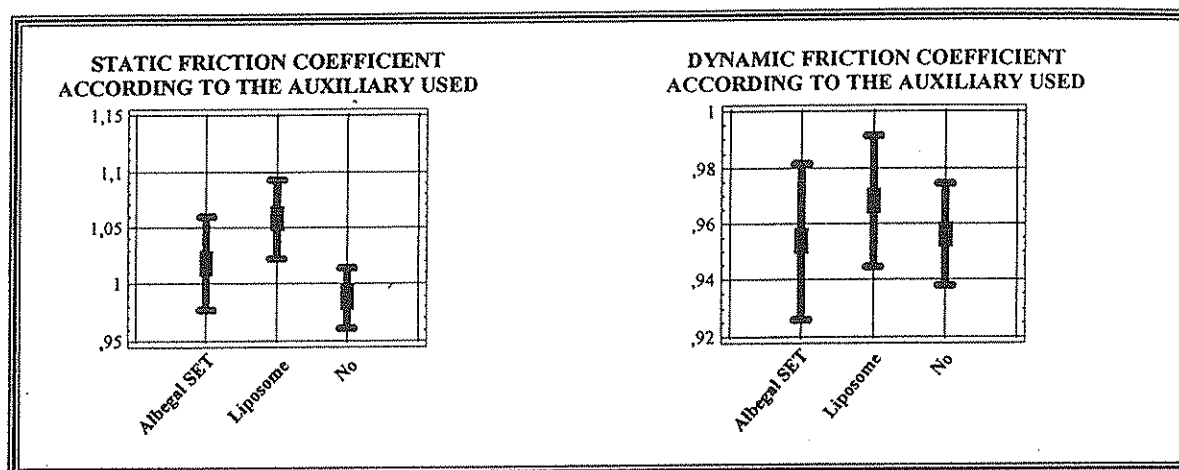
The use of liposomes as dye auxiliary seems to favours colour fastness. The results obtained on assessing staining for dyed samples using liposomes on the dyeing process indicates an slight improvement on colour fastness. Higher values are obtained on assessing staining over wool and polyamide fabrics compound compared to the other processes.

The handle improvement due to the presence of liposomes in the dyeing bath is also a great advantage important to be evaluated.

The handle characteristics of the wool dyed in the presence of liposomes was considered to be excellent by expert assessment, therefore, a series of objective measurements were performed. Figure 5 shows the influence of the auxiliary (Albegal SET, Liposome compared with dyeing made without auxiliary) on static friction. According to the ANOVA, the influence of the auxiliary on the

surface friction was significative at 10% level. Figure 5 shows the influence of the auxiliary on the surface dynamic friction. Although it seems that the liposome increases the dynamic surface friction, no significative differences were observed when the three different auxiliaries were used.

The higher values on static and dynamic friction obtained for samples dyed in the presence of liposomes are in accordance with the comfortable feeling assessed by the experts.



*Figure 5: Influence of the Dyeing Auxiliary on the Static Surface Friction Coefficient and on the Dynamic Friction Coefficient*

## CONCLUSIONS

From the results obtained in this study many advantages in the use of commercially available liposomes can be deduced, in front of other conventional auxiliaries.

Besides their competitive cost, in this study with Lanaset dyes, it can be concluded that the wool dyeing can be performed at 90°C using these liposomes with a satisfactory level of dye exhaustion and fixation as well as exhibiting a good fabric handle.

Furthermore, the use of liposome favours a retardant effect in the dyeing kinetic behaviour respect to the use of conventional auxiliary. All these facts conduct to a good dye levelling on the textile substrate.

Additionally the Lanaset dyes compatibility was confirmed by spectrophotometry. By this technique we can observe that the final colour obtained for both wool yarn samples was the same in the two dyeing processes (with liposomes or with Albegal SET).

Another positive characteristic of the liposome is the improving of fabric handle. The slight increase on the static surface friction induced by the liposome can be related with the more consistent and comfortable feeling assessed by the experts on the samples dyed with liposomes. Although the

influence on the dynamic surface friction was not significative, it seems that samples dyed with liposomes showed the highest level of friction.

In dyeing wool with Lanaset dyes there is a reduction of temperature, about 10°C, this entails an energy saving; besides there are ecological benefits of process due to the biological nature of the liposome used with a final better quality of the dyed textile.

## ACKNOWLEDGEMENTS

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